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(54) **PROCEDE DE PREPARATION D'UNE CELLULOSE
MICROCRISTALLINE DE GRANDE PURETE, EXEMPT DE
PRODUITS CHIMIQUES, A PARTIR D'UNE CELLULOSE
PRODUITE CHIMIQUEMENT**

(54) **PROCESS FOR PREPARING A HIGH PURITY
CHEMICAL-FREE MICROCRYSTALLINE CELLULOSE
FROM A CHEMICALLY PRODUCED CELLULOSE**

(57) On peut produire une cellulose microcristalline de grande pureté, exempte de produits chimiques, possédant un faible degré de polymérisation, au moyen d'un procédé dans lequel aucun acide tel que HCl, H₂SO₄ n'est nécessaire, comme c'est le cas dans les procédés d'hydrolyse acide classiques. Ledit procédé consiste à préparer une pâte à papier par retrituration, filtration et trituration d'une cellulose produite chimiquement. La pâte à papier ainsi produite est chauffée par vapocraquage, de sorte que de la pâte à papier traitée soit produite. Une force de cisaillement est appliquée sur la pâte à papier à la fin du vapocraquage. La pâte à papier traité est ensuite filtrée, lavée et blanchie, ce qui permet de produire de la cellulose microcristalline de grande pureté, exempte de produits chimiques. Grâce à ce procédé, on peut produire une cellulose microcristalline exempte de produits chimiques, en utilisant de la cellulose produite chimiquement, telle que de la cellulose kraft ou au bisulfite, comme matière de départ. La cellulose microcristalline de grande pureté obtenue selon ledit procédé peut être utilisée dans les industries pharmaceutiques et de la nutrition.

(57) A high purity chemical-free microcrystalline cellulose having a low degree of polymerization can be obtained by a process that does not require the use of any acids such as HCl, H₂SO₄ as are needed in conventional acid hydrolysis processes. This process comprises the steps of preparing a pulp by repulping, filtration and trituration of a chemically produced cellulose. The so-prepared pulp is subjected to heating through a steam explosion treatment in order to obtain a treated pulp. A shearing force is applied onto the pulp at the end of the steam treatment. The treated pulp is then filtered, washed and bleached thereby to obtain the high purity chemical-free microcrystalline cellulose. With this process, one may produce chemical free microcrystalline cellulose by using chemically produced cellulose such as kraft or bisulfite cellulose as starting material. The high purity microcrystalline cellulose obtained from the present process may be used in the pharmaceutical and nutritional industries.





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| (21) International Application Number: PCT/CA98/00479 (22) International Filing Date: 14 May 1998 (14.05.98) (71) Applicant (for all designated States except US): KEMESTRIE INC. [CA/CA]; 4245, rue Garlock, Sherbrooke, Québec J1L 2C8 (CA). (72) Inventors; and (75) Inventors/Applicants (for US only): JOLLEZ, Paul [CA/CA]; 2255, rue Vermont, Sherbrooke, Québec J1J 1G9 (CA). CHORNET, Esteban [CA/CA]; 470, Montmagny, Sherbrooke, Québec J1L 1T3 (CA). (74) Agent: ROBIC; 55, Saint-Jacques, Montréal, Québec H2Y 3X2 (CA). | (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published With international search report. | |
| (54) Title: PROCESS FOR PREPARING A HIGH PURITY CHEMICAL-FREE MICROCRYSTALLINE CELLULOSE FROM A CHEMICALLY PRODUCED CELLULOSE (57) Abstract <p>A high purity chemical-free microcrystalline cellulose having a low degree of polymerization can be obtained by a process that does not require the use of any acids such as HCl, H₂SO₄ as are needed in conventional acid hydrolysis processes. This process comprises the steps of preparing a pulp by repulping, filtration and trituration of a chemically produced cellulose. The so-prepared pulp is subjected to heating through a steam explosion treatment in order to obtain a treated pulp. A shearing force is applied onto the pulp at the end of the steam treatment. The treated pulp is then filtered, washed and bleached thereby to obtain the high purity chemical-free microcrystalline cellulose. With this process, one may produce chemical free microcrystalline cellulose by using chemically produced cellulose such as kraft or bisulfite cellulose as starting material. The high purity microcrystalline cellulose obtained from the present process may be used in the pharmaceutical and nutritional industries.</p> | | |

PROCESS FOR PREPARING A HIGH PURITY CHEMICAL-FREE
MICROCRYSTALLINE CELLULOSE FROM A CHEMICALLY PRODUCED CELLULOSE

FIELD OF THE INVENTION

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The present invention relates to a process for preparing high purity chemical-free microcrystalline cellulose having a low polymerization degree (DP) from a chemically produced cellulose.

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BACKGROUND OF THE INVENTION

Microcrystalline cellulose is obtained by depolymerization of cellulosic fibres down to their constitutive microcrystals.

15

Such a depolymerized cellulose is also known as level-off degree of polymerization cellulose (LODP).

20

The LODP accounts for the fact that cellulosic fibres are constituted of fibrils held together by significant amounts of disordered amorphous cellulose. The fibrils are mainly formed of highly ordered cellulose crystals. The latter are held together by adhesion and by small amounts only of amorphous cellulose in between the crystals.

25

The preparation of microcrystalline cellulose involves two very different steps:

(a) production of cellulose fibres; and

(b) transformation of the fibres into microcrystalline cellulose.

30

Existing processes for the production of microcrystalline cellulose start with the purchase of cellulose fibres and are followed by carrying out a conventional process such as hydrolysis or its variants to obtain the desired end product.

5

Such processes are not environmentally friendly since the cellulose fibres is derived from conventional processes using sulfur chemicals, such as SO_2 (sulfite process), Na_2S (Kraft process), and sulfuric acid (ethanol pulping process, or Allcell). Furthermore, the depolymerization of cellulose fibres to yield microcrystalline cellulose via an acid hydrolysis process uses mineral acids (HCl and H_2SO_4 being common acids).

10

Canadian patent No. 2,137,890 teaches the conversion of cellulose fibres (derived from conventional pulping processes) into microcrystalline cellulose by using benign reagents, namely O_2 and CO_2 . More particularly, this patent teaches that low degrees of polymerization, DP, may be obtained by a high pressure treatment of aqueous suspensions of cellulose with O_2 and CO_2 within autoclaves operated in batch mode. The treated product is subjected to centrifugal washing and spray drying to yield microcrystalline cellulose with a DP of about 300.

15

20

Canadian patent No. 2,139,400 discloses a process wherein a fibrous cellulose 2-10 wt% suspension is subjected to repeated passages through a high shear zone (two confronting surfaces, one fixed and the other having a rotational movement such as a double disk refiner) at temperatures not greater than 200°F . the obtained product is a cellulose having a high retention of water (about 350%). This cellulose is called "microdenominated cellulose".

25

30

Canadian patents No. 1,198,703 discloses a process for the simultaneous production of LODP microcrystalline cellulose and glucose by carrying out in a sequential manner using hardwoods and annual plants (such as straw and bagasse) as starting material. The separation of the hemicellulose, lignin and cellulose is accomplished via steam treatment (which liberates a significant fraction of the hemicelluloses), delignification (with benign organic solvents or alkaline solutions), and removal of residual hemicellulose from cellulose by an alkali treatment. The cellulose thus produced is soaked/impregnated with either HCl, H₂SO₄ or SO₂, and introduced into a reactor where saturated steam is added to reach 185°-240°C in less than 60 seconds. The treated material is withdrawn (i.e. expelled) from the reactor through a die by opening a valve. The expelled cellulosic solids are adiabatically quenched to 100°C in a fraction of a second. The product that is so obtained consist of disegregated cellulose fibrils (-20-50um in diameter and 1-2 mm in length) and glucose presumably produced from the hydrolysis of amorphous cellulose.

In view of the above, there would be an interest to develop an environmentally friendly process for producing a "green" (environmentally friendly) microcrystalline cellulose, which would use chemically produced cellulose such as a premanufactured specialty pulp as the starting material but would avoid the use of Cl- or S- containing acid reagents.

The Applicant has already proposed a process for producing chemical free cellulose which in turn may be used for producing microcrystalline cellulose. In this process, any suitable raw material such as softwoods, hardwoods, kenaf, bagasse and

straws may be used as starting material. The selected starting material is in the form of shavings, residues or sawdust are first subjected to heating under water vapour pressure for a certain period of time. Then, a violent depressurization is created thereby allowing an instant vaporization of the water from the wood which in turn causes the defibration of the wood matrix. The defibrated wood is then forwarded into a refiner and is subsequently soaked in water to wash away excess of hemicellulose. The lignocellulosic material obtained is then subjected to alkaline or alcohol extraction of the lignin. The latter can be further converted to phenolic products which can be used in the manufacture of adhesives for wood panels, or as raw material for pharmaceutical and nutritional products.

A purification step is then carried out for the purpose of concentrating and recovering the "good" fibres. The cellulose fibres so obtained are subjected to a complexation step. This step is necessary to eliminate the presence of ions that could interfere with the next step which is the bleaching of the cellulose.

After bleaching the cellulose, a fibrous cellulose is obtained. This fibrous cellulose undergoes a second steam explosion treatment, like the one previously described. This cellulose is finally bleached and microcrystalline cellulose is then recovered.

Thus, the prior process comprises the steps of:

a) mechanical and/or pneumatic purification of the shavings, residues or sawdust;

- b) primary steam explosion treatment (pressurization of the wood by steam);
- c) water extraction (for separating hemicellulose from the wood);
- 5 d) refining;
- e) alkaline or alcohol treatment (for extracting lignin);
- f) purification (for concentrating and recovering the "good" fibres);
- g) complexation (ion elimination);
- 10 h) bleaching of the fibrous cellulose that is so obtained;
- i) secondary steam explosion treatment, and
- j) bleaching of the fibrous cellulose.

15 The Applicant has now found a way to produce a high purity chemical free microcrystalline cellulose having a low polymerization degree by using a chemically produced cellulose.

SUMMARY OF THE INVENTION

20 Thus, it is an object of the present invention to provide a new process for producing microcrystalline cellulose having a low degree of polymerization (DP) by using a chemically produced cellulose (kraft, sulfite) as starting material.

25 More particularly, the object of the present invention is to provide a process for preparing microcrystalline cellulose by using as a starting material, a chemically produced cellulose which may be in the form of a premanufactured specialty pulp. This process comprises the steps of:

- a) preparing a pulp by repulping, filtration and trituration of the chemically produced cellulose;
- b) subjecting the pulp obtained in step a) to a steam explosion treatment in order to obtain a microcrystalline cellulose;
- 5 c) filtering and washing the microcrystalline cellulose;
- d) bleaching the microcrystalline cellulose obtained in step c) to obtain a bleached microcrystalline cellulose; and
- e) filtering and washing the bleached microcrystalline cellulose of step d) to obtain the high purity chemical free
- 10 microcrystalline cellulose.

In accordance with the invention, the process is characterized in that at the end of the steam explosion treatment of step b), a shearing force is applied onto the pulp.

15

Thus, it has been found a way to depolymerize cellulose to low DPs by treating a cellulose containing aqueous suspension via an appropriately designed floating fixed head high shear devices such as a valve, near the glass transition temperature

20 of cellulose, typically in the 230°-240°C range. The shearing force applied on the pulp is the result of the combined effect of instant depressurization and sudden flow of the pulp through a discharge valve.

25 Hence, through this process, it is possible to transform chemically produced celluloses (Kraft, sulfite), via water soaking/impregnation, and aqueous/steam treatments using only water (liquid and steam), into low DP microcrystalline cellulose.

30

As can be appreciated, the process of the invention comprises a bleaching step (d) where the microcrystalline cellulose is bleached by using benign chemicals such as caustic and hydrogen peroxide. Hence, the process of the present invention does not
5 require the use of acid. Nonetheless, classical bleaching reagents may be used for bleaching the microcrystalline cellulose if desired.

The process of the present invention allows the production of
10 microcrystalline cellulose having a DP comprised between 150 and 260, a whiteness higher than 80 and a cristallinity index of about 83% to 87%.

DETAILED DESCRIPTION OF THE INVENTION

15

In the process of the present invention, a premanufactured pulp, such as a specialty pulp, which is a bleached cellulose from angiosperm or gymnosperm obtained from kraft, bisulfite chemical processes or any conventional pulping processes, is
20 used as the starting material.

Softwoods provide microcrystalline cellulose with a level of DP (LODP) >200, whereas hardwoods and straws yield microcrystalline cellulose having level of DP(LODP) <200. The
25 degree of polymerisation can be tailored and optimized that is reduced by subsequent aqueous/steam treatments. However, this will cause a reduction in the final yield of the microcrystalline cellulose.

30 When carrying out the process of the invention, the starting material must not be in a condensed state so that it is easily

transported. It must contain a residual humidity varying from 35 to 65%. Hence, if the starting material is a premanufactured pulp, it must be repulped in order to increase its humidity and to render it in a less condensed state. This in turn will increase its reactivity.

The cellulose having a proper level of humidity is subjected in a reactor to a steam explosion treatment in a continuous or discontinuous batch mode.

10

The steam explosion treatment is carried by pressurization of the reactor. This is done by injecting steam in the reactor. The various conditions such as temperature, pressure and residence time are perfectly determined and controlled so that it is possible to develop a mathematical model, referred to as the "severity" factor, R_0 , for determining the final quality of the treated cellulose.

15

"Severity" stands for a hydrolysis control parameter represented by the symbol R_0 and expressed by the mathematical formula which combines two variables, namely the time of heating and the temperature or vapour pressure.

20

$$R_0 = t \times e^{T-100/14.75}$$

25

t is the time of heating in minutes and T is the temperature in Celsius.

30

A shearing force is applied at the end of the steam explosion treatment and the treated material is subjected to filtration. The shearing force is the result of the combined effect of a violent vaporization induced by an instant depressurization

and a sudden flow of the pulp out of the reactor through a discharge valve. The cellulose is simultaneously washed in order to prepare the pulp for bleaching.

5 During the steam treatment, it is recommended to work at a pressure varying from 215 to 440 psig, at a temperature varying between 200° and 240°C, and at a contact time varying from 4 to 24 minutes.

10 As mentioned above, an important aspect of the present invention lies in that at the end of the steam explosion treatment, a shearing force is applied on the treated pulp.

The final bleaching using sodium hypochlorite at 40°C, pH > 9
15 during 1 or 2 hours or hydrogen peroxide is carried out at a pH comprised between 10 and 11, at a temperature of 60°C or 110° to 120°C under nitrogen or oxygen pressure varying from 60 to 120 psi, and in the presence of magnesium sulfate and sodium silicate.

20

EXAMPLE 1

(I) PREPARATION OF THE POPULUS TREMULOIDES SOLUBLE PULP

The method to prepare this type of pulp comprises a step of
25 steam explosion which could be carried out in a continuous or discontinuous batch mode. The pressure, temperature and contact time are perfectly determined and controlled, since they have been modeled for each type and form of biomass and each type and size of reactor.

30

The poplar chips (6 mm of thickness and 1 to 2 inches in width) must contain a humidity level of between 35% and 65% calculated as base humidity.

5 Their dry composition are the following:

| | |
|-------------------|---------|
| Total extract : | 5, 03% |
| Alpha cellulose : | 44, 92% |
| Holocellulose : | 77, 30% |
| Pentosans : | 17, 82% |
| 10 Lignin : | 17, 67% |
| Ash : | 0, 19% |
| DP : | 1352 |

15 A 6 litre reactor is charged with the wood and is supplied by steam. The reactor is maintained at a pressure of 380 psi for 3 minutes before carrying out instant depressurization. The resulting pulp from the cyclone is refined at a pulp consistency of 2% for 90 seconds at 18000 rpm in a Waring Blender Commercial Model 38 BL.19 with water at 60°C or up, so
20 as to eliminate the hemicellulose liberated during cooking.

The lignocellulose obtained is mixed in a solution of sodium hydroxide at 22 grams per litre so as to acheive a pulp consistency of 10%. The treatment is carried out at 160 °C for
25 90 minutes.

After washing, the partially delignified cellulose has a kappa index between 6 and 8. A purification step of the pulp allows to separate fines and other impurities so to obtain a very low
30 kappa index which is between 3 and 5.

The cellulose is then bleached either by the classical sequences of bleaching CepHD:

-chlorination at $\text{pH} < 2$ with 0,08 g/l of active chlorine at a temperature of 25°C

5 -extraction of chloro-lignin by washing the fibres at 40°C/by means of a hydroxide sodium solution at 3 g/l and containing hydrogen peroxide at 0, 2 g/l

-hypochlorination at 40°C, 0,3 g/l of active chlorine at a temperature of 70°C and a pH varying from 3,5 to 7,0.

10

The bleached cellulose is obtained with an output of 35% on the original chips (output calculated from the dry bases). and embodies the following characteristics:

S 10 : 4, 89%

15 S 18 : 2, 26%

Pentosans : 1, 27%

Alpha cellulose: 96, 40%

Ash : 0, 10%

DP 787

20

This high purity cellulose may be used as a soluble pulp in the production of viscose, cellulose acetate and microcrystalline cellulose.

25

(II) PROCESS FOR PREPARATION OF MICROCRYSTALLINE CELLULOSE

The bleached cellulose pulp obtained herein above is transformed into microcrystalline cellulose by a process which is an object of the present invention.

30

5 The soluble pulp is brought to a residual humidity comprised between 35% and 65%, preferably 50% before being treated with steam at 350 psi for 8 minutes, washed and bleached with hydrogen peroxide at pH 10,5 during 60 minutes at 60°C and preferably at 120°C under nitrogen or oxygen pressure.

The so obtained microcrystalline cellulose has the following characteristics:

DP: 170

10 Crystallinity index (RX) : 87%

EXAMPLE 2

(I) PREPARATION OF CELLULOSE PULP FROM SUGARMAPLE SAWDUST

15 The cellulose of this example was prepared from selected sugarmaple sawdust. The pulping process is similar to the one described in example 1, wherein the heating step is carried out at a pressure of 350 psi for 4 minutes. The purification step is maintained so as to have a pulp consistency of 2%. The
20 lignocellulose is treated at a pulp consistency of 12% in a sodium hydroxide solution (28g/l) at 160°C for 90 minutes. The lignin is solubilized in the form of sodium lignate.

25 The cellulose so obtained has a kappa index of 10. A chelating agent is added to the treatment so as to prepare the bleaching with hydrogen peroxide (2% from dry base) at pH 10,5 in a pulp consistency of 12%. After washing and filtration, the cellulose comprises the following characteristics:

30 DP 700

Pentosans 1,20%

Holocelluloses: 98,05%

Ash: 0,70%

kappa index: 3

5 This cellulose may be used as a food fibre due to its high purity and its method of production which do not require any use of chlorinated and/or sulphurated products. This cellulose may also be used as a starting material for the application of specialty pulp production, viscose, cellulose acetate, etc.

10

(II) PROCESS FOR PREPARING THE MICROCRYSTALLINE CELLULOSE

The cellulose obtained herein above is subjected to a reactor for the steam explosion treatment. This treatment is carried out at 320 psi for 8 minutes. After this treatment, an instant depressurization is carried out in the reactor. A bleaching step is carried out and the resulting microcrystalline cellulose embodies the following characteristics:

15

DP : 182

20

Crystallinity index (RX) : 85

Density: 0, 28g/cm³

Ash: 0, 10%

The output of this process on the initial cellulose is 80%.

25

EXAMPLE 3

Cellulose HV 20* from sulphite pulp production from softwoods having the following characteristics was used as the starting material:

30

* Trade-marks

S 10 at 25°C : 5,8%

S 18 at 25°C : 5,2%

Intrinsic viscosity : 14,46 dl/g

Pentosans : 4,4%

5 Ash: 0,17%

Whiteness: 87,5%

DP 1966

10 The treatment that was applied for a pulp having a high DP was
16 minutes under 320 psi. The obtained cellulose was sieved on
70 mesh. This cellulose can be bleached at 60°C using 2% of
hydrogen peroxide in the presence of magnesium sulfate and
sodium silicate at pH 10,5. In the case where it is bleached
15 with sodium hypochlorite 1% on dry base, it is carried out at
40°C for 2 hours with a pulp consistency of 10% and at a pH >9.
The output of these operations is 80% on dry cellulose as the
starting material.

20 The so obtained microcrystalline cellulose has the following
characteristics:

DP 253

Cristallinity index (RX): 85%

Ash : <500ppm

25

EXAMPLE 4

Cellulose Q90*, a resinous kraft pulp (20% Jack pine, 80% Black
Spruce) having the following characteristics was used as a
starting material for the present example.

30

*Trade-marks

Viscosity : 21,4 cps

Whiteness Iso : 89%

DP : 1156

5 Two consecutive treatments of 8 minutes at 320 psi were carried out. After sieving on 70 mesh and bleaching with hydrogen peroxide 2%, the microcrystalline cellulose embodied the following characteristics:

10 DP : 246

Cristallinity index (RX): 83%

Ash: <500 ppm

15 Although the present invention has been explained hereinabove by way of a preferred embodiment thereof, it should be understood that the invention is not limited to this precise embodiment and that various changes and modifications may be effected therein without departing from the scope or spirit of the invention.

CLAIMS

1. Process for preparing a high purity chemical-free microcrystalline cellulose having a low degree of polymerization, comprising the steps of

a) preparing a pulp by repulping, filtration and trituration of a chemically produced cellulose;

b) subjecting the pulp obtained in step a) to a steam explosion treatment in order to obtain a microcrystalline cellulose;

c) filtering and washing the microcrystalline cellulose;

d) bleaching the microcrystalline cellulose of step c) to obtain a bleached microcrystalline cellulose; and

e) filtering and washing the bleached microcrystalline cellulose of step d) to obtain the high purity chemical-free microcrystalline cellulose,

the process being characterized in that at the end of the steam explosion treatment of step b), a shearing force is applied onto the pulp without the addition of any chemical agent.

2. The process according to claim 1, characterized in that the chemically produced cellulose is a bleached cellulose from angiosperm or gymnosperm.

3. The process according to claim 2, characterized in that the bleached cellulose is obtained from a kraft process.

4. The process according to claim 2, characterized in that the bleached cellulose is obtained from a bisulfite process.

5. The process according to claim 2, characterized in that the bleached cellulose is obtained from a conventional steam explosion process.

5 6. The process according to any one of claims 1 to 5, characterized in that the steam explosion treatment of step b) is carried out in a continuous or discontinuous manner.

10 7. The process according to claim 6, characterized in that the steam explosion treatment of step b) is carried out in a continuous manner.

15 8. The process according to any one of claims 1 to 7, characterized in that the steam explosion treatment of step b) is carried out under a pressure of from 215 to 440 psig and at a temperature of from 200° to 240°C.

20 9. The process according to any one of claims 1 to 8, characterized in that the shearing force is applied near a glass transition temperature of cellulose comprised between 230° and 240°C.

25 10. The process according to any one of claims 1 to 9, characterized in that the steam explosion is carried out in a reactor with a discharge valve and the shearing force is applied on the pulp by instant depressurization and sudden flow of the pulp through the discharge valve.

30 11. The process according to any one of claims 1 to 10, characterized in that in step d), the microcrystalline

cellulose is bleached with a caustic and hydrogen peroxide under nitrogen pressure.

12. The process according to claim 11, characterized in that the step d) is carried out at a temperature of about 110 to 120°C and that the nitrogen pressure varies from 60 to 120 psi.

13 A microcrystalline cellulose obtained by the process according to any one of claims 1 to 12, wherein said microcrystalline cellulose has the following characteristics:

- a degree of polymerization ranging between 150 and 260 ;
- a whiteness >80; and
- a cristallinity index ranging between 83 and 87%.